--- 5/00246

10/518037

REC'D 3 0 APR 2003

WIPO PCT



Kongeriget Danmark

BEST AVAILABLE COPY

Patent application No.:

PA 2002 00907

Date of filing:

14 June 2002

Applicant:

Olicare ApS

(Name and address)

Islevdalsvej 187

2610 Rødovre

Denmark

Title: A PROCESS AND AN APPARATUS FOR PURIFICATION OF MINERAL OIL.

IPC: B 01 D 39/04; B 01 D 39/06

This is to certify that the attached documents are exact copies of the above mentioned patent application as originally filed.



Patent- og Varemærkestyrelsen Økonomi- og Erhvervsministeriet PRIORITY DOCUMENT

SUBMITTED OR TRANSMITTED IN COMPLIANCE WITH RULE 17.1(a) OR (b)

24 April 2003

Ro 7 Tidomonn



1

Modtaget PVS 14 JUNI 2002

A process and an apparatus for punification of mineral oil

Technical field of the invention

5 The present invention concerns a process and an apparatus for the purification of mineral oil

Background of the invention

After the re-refining of waste oil an oil product containing several waste materials is obtained. The waste materials which comprise several residues from additives and from oxidation of oil in the re-refining process include hydrogen sulfide and soot giving the oil a dark colour and a strong and unpleasant smell. Conventionally the oil product is used as low budget fuel which can cause environmental problems. It is therefore desirable to remove the waste materials from the oil.

The following patents are dealing with the purification of oil by using filters

20 US patent nr 4988440 discloses a filtering unit for cleaning hot cooking oil. The filtering unit contains activated carbon, calcium and/or magnesium silicate, cellulosic fiber and a binder. The hot cooking oil is decolorized and odour causing components are adsorbed by using the filter. The purpose of the silicates is to remove free fatty acids from the oil.

US patent nr 6321915 discloses a filter media which comprises a blend of activated carbon containing inorganic fibers, inorganic fiber whiskers and a binder

The filtermedia can be used for removing unwanted species and particles from industrial oils. The inorganic fibers, however, have shown not to be useful for removing smelly components and colour.

Filters as described in US patent no 4988440 and US patent no 6321915 would not be useful for purifying mineral oil since because of a fast blocking of the filters with necessary often change of the filter cartridge as a consequence

5

10

The object of the invention is to present a process by which mineral oil is purified into a product devoid of smell and colour and by which process problems concerning blocking of the filters are reduced. The object is also to provide a process which can be carried out in a continuous way resulting in a continuous flow.

The object of the invention is furthermore to present an apparatus which is useful in the process

15 Summary of the invention

Mineral oil which has been used for example in automobile industry or in other industries is traditionally treated in a re-refining process. The remaining oil product after the re-refining process has a high content of particles giving a dark colour to the oil and it also has a strong smell. The oil product is used as a low-budget fuel which, because of the content of waste materials, can cause environmental problems in the burning process of the fuel. It is therefore desirable to purify this product in order to avoid the environmental problems.

25

20

The object of the invention is solved by a process comprising the steps of

prefiltrating the oil followed by

30

 passing the prefiltrated oil through a filtering unit in which the filtermedia comprises organic fibres and carbon particles, said fibres and carbon particles being adhered to each other by a binder

___2_6________

3

According to the process mineral oil is purified and the product obtained by using the process is a basic (virgin) oil. Hydrogen sulfide and residues from carbonization are effectively removed.

By prefiltrating the oil most of the soot particles are removed. The prefiltrated oil is passed through a filtering unit in which the rest of the particles and also substances causing odour are removed resulting in a basic (virgin) oil product. Because of the prefiltrating treatment the lifetime of the last filtering unit is prolonged and a relatively more effective removal of the substances causing odour is obtained. This is due to the fact that a relatively larger part of the carbon particles in the filter can be used in the removal of the substances causing odour and colour.

Brief description of the drawing

15

The drawing shows a flow diagram of a preferred embodiment of the invention

Detailed description of the invention

20

25

The oil to be purified contains particles and smelly components. The particles give the oil a dark colour and the smelly components gives the oil a strong rotten and burnt smell. The product has so far been considered a kind of waste product only useful as a fuel. Still the product has a content of a valuable raw material. By the process according to the invention the particles and the smelly components are separated from the oil and the raw material, basic (virgin) oil, is obtained.

The prefiltration of the oil is essential to achieve an optimal effect in the following filtering unit as mentioned above and also to achieve a process with a continous flow

15

20

4

The prefiltration of the oil can be carried out in one or more steps. In a preferred embodiment the prefiltration is carried out in more steps. This has the effect of increasing the overall prefiltering effectivity since the probability of trapping a particle is increased by using more filters. It is furthermore an option to choose the prefiltration units so that they trap particles with decreasing sizes in the direction of the flow. This results in an extended lifetime of the prefilters because the period of time until blocking occurs is prolonged providing a better economy for the process.

One prefiltration step may be used in situations with particles of a known uniform size

In one embodiment of the invention the oil is prefiltrated by passing through three prefiltration units. Depending on the sizes of the particles in the oil the filters that trap particles of the relevant size can be chosen. For example the prefiltration units can have the following characteristics. A first prefiltration unit trapping particles with a diameter bigger than the order of magnitude of 12 μ m, a second prefiltration unit trapping particles with a diameter bigger than 6 μ m and a third prefiltration unit trapping particles with a diameter bigger than 1 μ m. This embodiment has shown to result in a very good economy for the process. The filtering material in these units may be made of different kinds of fibres for example glass fibres and fibres made of polymenc materials.

The filtering unit has a filtermedia comprising organic fibres, carbon particles and a binder. This type of filtermedia has shown to result in a very high degree of purification towards smell and colour in mineral oil.

Mineral oil comprises any class of oils that are of mineral origin

30

The term "binder" comprises any material which is capable of holding the fibres and the carbon particles together with adhesive forces. For example the binder can be a resinous compound. Preferably the binder is a positively

charged resin. A resin comprises any semi-solide or solid organic compound or mixture of organic compounds being sticky at certain temperatures. The positively charged resin gives a positive charge to the fibre material implying a more effective attraction of the waste materials.

5

10

20

25

30

By organic fibres is meant fibres originating from naturally occurring materials or fibres of synthetic polymeric materials. By the term "natural" is meant any fibre that originates from plant materials. Cellulosic fibres originating from wood, cotton or linen are suited natural fibre materials and filter medias consisting of cellulosic fibers carbon particles and a binder has shown to give a very high degree of purification. By synthetic fibres is meant any fibre which is synthetically produced. The synthetic fibres include fibres from polymeric material.

15 Carbon particles include carbon in crushed, pulverized, powderized form or carbon in any other particle-like form

The filter media can be produced by mixing carbon particles, fibres and binder followed by stirring in deionized water until a suitable consistency is achieved. Then the material is poured on a grating to drain off the water and the material is equally distributed by vibrating and compressing. The material is punched in the desired form, for example in a circular form, and is now a filtering plate ready for use. By plate is meant a piece of material of which the thickness is small compared to the length and width. The filter media is optionally equipped with a net of supporting material. Such a filtering plate is easy to handle and the replacement of the plates is easily done as well. By a net is meant any reticulated piece of material. The purpose of the net is to hold back the material of the filter media if relatively high pressure is applied. For example, the net is made of a polymeric plastic or of steel. A net of polymeric plastic has the advantage of the entire filter media capable of being burnt after use.

6

In another preferred embodiment of the invention which is particularly useful when the oil contains free gases after passing through the prefiltration units and before being passed through the filtering unit, the oil is to pass through one or more vacuum units. In these units subatmospheric pressure exists and free gases are liberated. In the filtering unit the residual waste products can be even more effectively removed since the free gases are already removed and thus does not take up capacity in the filtering unit.

Preferably the oil before being prefiltrated is heated to a temperature of 50-90 °C. The heating results in lowering the viscosity of the oil which may help keeping a high lifetime of the prefiltrating filters. If the starting oil is a low-viscosity oil, said heat treatment is not necessary. Another effect of the heating is that any free gases are released more easily in the vacuum units.

15 If the oil is in a heated condition when it reaches the filtering unit it is advantageous to pass the oil through a cooling unit before the end of the purification Preferably the cooling unit is placed immediately before entering the filtering unit. The cooled oil is of a higher viscosity and the retention time in the filtering unit is therefore extended causing a more effective adsorption of the smelly components to the carbon particles in the filter. Preferably the oil is cooled to a temperature of 10-30 °C.

After passing the filtering unit the oil may optionally be passed through an additional filter unit. The purpose of this unit is to trap any residues of carbon escaping from the filtering unit.

A preferred embodiment of the process is shown in the drawing. The letters of the drawing refer to the following steps

30 A Pump

- **B** Heater
- C Prefiltration
- D Vacuum towers

7

E Cooler

F Filtering unit

G Additional filter

It is to be understood that only C and F are essential step, the rest is optional to be decided in accordance with the specific purification problem

The oil is pumped by a displacement pump (A) equipped with a check valve in order to regulate the pressure. The oil is optionally passing through a heater (B) in which the oil is heated to a temperature of 50-90°C resulting in a low-viscosity oil prolonging the lifetime of the filters in the following prefiltration steps.

The heated oil is now passed to the prefiltration step (C) In the embodiment of the invention shown on the drawing the prefiltration step comprises three prefiltration units. Each unit may be supplied with a by-pass valve. The first unit traps the particles bigger than the order of magnitude of 12 µm, the second unit traps the particles bigger than 6 µm and the third unit traps particles bigger than 1 µm. In this way the lifetime of the filtermedia in the prefiltrating units is extended.

Any number of prefiltration filters and sizes of the voids in the filters may be chosen in accordance with the specific purification problem

- 25 From the prefiltration step the oil is passed on to three vaccum towers (D) Each unit is supplied with a by-pass valve. In the vacuum towers any free gases is released resulting in an even more effective removal of waste materials in the following filtering unit.
- Optionally the oil is sent through a cooling step (E) giving a high-viscosity oil The high-viscosity oil has a longer retention time in the following filtering unit providing a more effective removal of the waste products. From the cooler the oil is passed on to the filtering unit (F). In this unit the filtering media

comprising cellulosic fibres and carbon particles being adhered to each other by a positively charged resinous binder are removing waste materials including odour- and colour-causing components. The unit may be supplied with a by-pass valve

5

The oil is now optionally passed on to an additional or security filter (G) In this filter any carbon material from the filtering unit that has been detached is trapped. The filter is supplied with a security valve. The product obtained by the process is a purified product devoid of smell and colour.

10

The following example illustrates the invention in a preferred embodiment

Example 1

A re-refined waste oil was purified by passing the oil through the treatment steps shown on the drawing. The oil was passed by the pump (A) through the heater (B) and from the heater the oil was passed through three prefiltration units (C). After this the oil was passed through three vacuum towers (D). Subsequently the oil was cooled in the cooler (E). The cooled oil was treated in the filtering unit (F). Finally the oil was passed through an additional filter (G). The content of particles, the colour level and the smell of the oil were measured/observed at the entrance, after the prefiltration units, after the vacuum towers and after the filtering unit. The following results were obtained.

9

| | | Entrance | After prefiltration | After vacuum towers | After filtering unit |
|--|----|-------------------------------------|------------------------|--|----------------------|
| Level particles (mean values) | of | 21/19/17 or less | 14/11/9 or less | 14/11/9 or less | 14/11/9 or less |
| Level colour (mean values) | of | 3.5-4 or less | 3.2 or less | 3,2 or less | 0,5-1 or less |
| Smell | | Strong rotten and burnt smell | _ | Less strong rotten and burnt smell | |

The numbers of particles are measured by using an automatic lazer particle counter of the type Met-one / Hiac Royco. The particle level is determined as specified in the international standard ISO 4406. This standard relates the numbers of particles to a level of contamination by particles. The three numbers refer to the level of particles of with a diameter bigger than 2 μ m, 5 μ m and 15 μ m respectively

The colour level is measured as specified in ISO 2049. The standard defines a method for the visual determination of the colour of oil products. For the determination is required a sample in a container and a colorimeter. The container is placed in the colorimeter and a light source is switched on to illuminate the sample. The sample is now compared to colour standards. It is determined which standard matches best the colour of the sample. The result is noted as an identification number of the standard matching the sample best.

The smell has been subjectively judged by shaking the sample of oil, removing the lid of the sample container and smelling the sample of oil

5

From the example it appears that the oil is freed from smelly components and practically colourless

Modtaget PVS

Patent Claims

- 1 A process for purification of mineral oil comprising the steps of
- 5
- prefiltrating the oil followed by
- passing the prefiltrated oil through a filtering unit in which the filtermedia comprises organic fibres and carbon particles, said organic fibres and carbon particles being adhered to each other by a binder

10

- 2 A process according to claim 1 in which the oil is prefiltrated by passing through one or more prefiltration units
- 3 A process according to claim 2 in which the oil is prefiltrated by passing it through more prefiltration units
 - 4 A process according to claim 3 in which the prefiltration units remove particles with decreasing sizes in the direction of the flow
- 5 A process according to any of claims 3-4 in which the oil is prefiltrated by passing it through three prefiltration units
- 6 A process according to claim 5 in which the first prefiltration unit is trapping particles bigger than the order of magnitude of 12 μm, the second prefiltration unit trapping particles bigger than 6 μm and the third prefiltration unit trapping particles bigger than 1 μm
 - 7 A process according to any of claims 1- 6 in which the prefiltration is performed by using a filtering media made of glass fibres

30

8 A process according to any of claims 1-7 in which the filtering media in the filtering unit contains 5-95% carbon based on the weight of carbon particles and fibres

25

- 9 A process according to any of claims 1-8 in which the fibres in the filtering unit are natural fibres
- 5 10 A process according to claim 9 in which the fibres are cellulosic fibres
 - 11 A process according to any of claims 1-10 in which the binder is a positively charged resin
- 10 12 A process according to any of claims 1-11 in which the fibres, the carbon particles and the binder is in the form of a filtering plate
 - 13 A process according to claim 12 in which the filtering plate is supported by a net
 - 14 A process according to claim 13 in which the filtering plate is supported by a net of plastic
- 15 A process according to claim 13 in which the filtering plate is supported by a net of steel
 - 16 A process according to any of claims 1-15 in which the oil after being passed through the prefiltration units and before being passed through the filtering unit is passed through one or more vacuum units
 - 17 A process according to any of claims 1-16 in which the oil before being passed through the prefiltration units is heated to a temperature of 50-90 °C
- 18 A process according to any of claims 1-17 in which the oil is cooled 30 immediately before passing through the filtering unit
 - 19 A process according to claim 18 in which the oil is cooled to a temperature of 10-30 °C

 C_{k}

13

20 A process according to any of claims 1-19 in which the oil after being passed through the filtering unit is passed through an additional filtration step

5

- 21 A process according to any of claims 1-20 in which the oil is forced through the treatment steps by the use of a pump
- 22 An apparatus for the purification of mineral oil comprising

10

- means for prefiltrating the oil and
- a filtering unit in which the filtering media comprises organic fibres and carbon particles said organic fibres and carbon particles being adhered to each other by a binder

15

- 23 An apparatus according to claim 22 in which the filtering media in the filtering unit contains 5-95% carbon based on the weight of carbon particles and fibres
- 24 An apparatus according to any of claims 22-23 in which the fibres in the filtering unit are natural fibres
 - 25 An apparatus according to claim 24 in which the fibres are cellulosic fibres

- 26 An apparatus according to any of claims 22-25 in which the binder is a positively charged resin
- 27 An apparatus according to any of claims 22-26 in which the fibres, the carbon particles and the binder are in the form of a filtering plate
 - 28 An apparatus according to claim 27 in which the filtering plate is supported by a net

- 29 An apparatus according to claim 28 in which the net is made of plastic
- 30 An apparatus according to claim 28 in which the net is made of steel

5

- 31 An apparatus according to any of claims 22-30 in which the prefiltration means comprise one or more prefiltration units
- 32 An apparatus according to claim 31 in which the prefiltration means comprise more prefiltration means
 - 33 An apparatus according to claim 32 in which the prefiltration units remove particles with decreasing size in the direction of the flow
- 15 34 An apparatus according to any of claims 32-33 in which the prefiltration means comprise three prefiltration units
- 35 An apparatus according to claim 34 in which the first unit is trapping particles bigger than approximately 12 μm, the second prefiltration unit is trapping particles bigger than approximately 6 μm and the third prefiltration unit is trapping particles bigger than approximately 1 μm
 - 36 An apparatus according to any of claims 22-35 in which the prefiltrating means comprise filters with a filtermedia made of glass fibres

- 37 An apparatus according to any of claims 22-36 said apparatus comprising one or more vacuum units said vacuum units being placed in the direction of the flow immediately after the prefiltrating means
- 30 38 An apparatus according to any of claims 22-37 in which a heater is placed in the direction of the flow immediately before the prefiltrating means

- 39 An apparatus according to any of claims 22-38 in which a cooler is placed in the direction of the flow immediately before the filtering unit
- 40 An apparatus according to any of claims 22-39 comprising an additional filter said filter being placed in the direction of flow after the filtering unit
 - 41 An apparatus according to any of claims 22-40 comprising a pump

Modtaget PVS 14 JUNI 2002

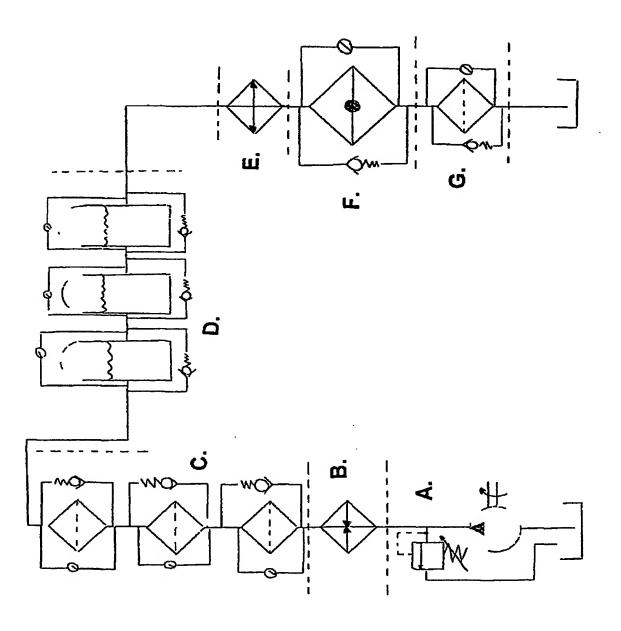
A process and an apparatus for purification of mineral oil

ABSTRACT

The present invention concerns a process and an apparatus for the purification of mineral oil. The process comprises the steps of prefiltration of the oil followed by passing the prefiltrated oil through a filtering unit in which the filtermedia comprises organic fibres and carbon particles, said organic fibres and carbon particles being adhered to each other by a binder. By performing the process mineral oil is purified and hydrogen sulfide and residues from carbonization are effectively removed. Problems concerning blocking of the filters are reduced and the process is carried out in a continuous way resulting in a continuous flow.

Modtaget PVS 14 Jun. 2002

Fig. 1



This Page is Inserted by IFW Indexing and Scanning Operations and is not part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

| Defects in the images include but are not limited to the items checked: |
|---|
| BLACK BORDERS |
| IMAGE CUT OFF AT TOP, BOTTOM OR SIDES |
| FADED TEXT OR DRAWING |
| BLURRED OR ILLEGIBLE TEXT OR DRAWING |
| SKEWED/SLANTED IMAGES |
| COLOR OR BLACK AND WHITE PHOTOGRAPHS |
| GRAY SCALE DOCUMENTS |
| ☐ LINES OR MARKS ON ORIGINAL DOCUMENT |
| ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY |
| OTHER: |

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.